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Filter Extraction Procedures and Results for Various EPA/ECTD (Environmental Protection Agency/Emission Control Technology Division) Particulate Samples

Southwest Research Inst., San Antonio, TX

Prepared for

Environmental Protection Agency, Ann Arbor, MI

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by

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Contract No. 68-03-2884
Task Specification 9

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Prepared for

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Emission Control Technology Division
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FOREWORD

This project was conducted for the U.S. Environmental Protection Agency by the Department of Emissions Research, Southwest Research Institute. The work was carried out between April 1981 and June 1982 under EPA Contract No. 68-03-2884, Task Specification Number 9. It was identified within Southwest Research Institute as Project 05-5830-009. The scope of work defined by EPA is located in Appendix A of this report. The EPA Project Officer was Mr. Robert J. Garbe, and the Task Technical Officer was Mr. Thomas M. Baines, both of the Characterization and Technical Applications Branch, Emission Control Technology Division, Environmental Protection Agency, 2565 Plymouth Road, Ann Arbor, Michigan. The Southwest Research Institute Project Manager was Charles T. Hare, and the Project Leader and Principal Investigator was Mary Ann Warner.

ABSTRACT

This report describes filter extractions and benzo(a)pyrene analyses performed for the Emission Control Technology Division of the Environmental Protection Agency. Pallflex filters measuring 20x20 inches were soxhlet-extracted in methylene chloride to remove organic soluble material. Some of the extracts were analyzed for benzo(a)pyrene, and some underwent Ames bioassay. Percent extractables and benzo(a)pyrene concentrations in the extracts are reported. Part of the effort also went into splitting and recombining extracts with the necessary extra drying and weighing steps.

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I. INTRODUCTION

The purpose of work directive number 9 of EPA Contract No. 68-03-2884 was to perform soxhlet extractions and BaP analyses on diesel particulate samples collected from various sources. Selected samples were also to be sent to Microbiological Associates (formerly EG&G Mason Research Institute) for Ames bioassay. The initial scope of work (found in Appendix A) called for the extraction of 239 filters, analysis of 49 samples for benzo(a)pyrene, and the shipment of 82 extracts for Ames bioassay. This was altered during the program at the request of the Project Officer. A total of 146 filters were extracted (37 from EPA, 109 from Southwest Research Institute's Emissions Laboratory), 21 extracts were analyzed for benzo(a)pyrene, and 17 were shipped for Ames bioassay. As a substitution, extra sample work-up involving the combination and splitting of extracts was performed. Additionally, extracts from 52 pre-extracted filters were combined to form aggregate samples. Tables 1 through 5 in Section III describe the treatment of each extract or filter during sample processing.

II. ANALYTICAL PROCEDURES AND INSTRUMENTATION

Particulate-loaded filters were extracted in a soxhlet apparatus with methylene chloride as the solvent. The extracts were filtered, concentrated under vacuum, transferred to preweighed glass vials, and weighed. Whenever possible, two filters were extracted in one soxhlet. Some extracts were also combined to form composite samples, and subsequently split up as required. Several extracts were analyzed for the presence of benzo(a)pyrene using a high pressure liquid chromatograph. The method closely follows the analytical procedure developed by General Motors Research Laboratories for measuring benzo(a)pyrene in organic extracts of automotive exhaust.(1) The analytical system consists of a Perkin-Elmer Series 2 Liquid Chromatograph, Model 650 Fluorescence Detector, and Model 150B Xenon Power Supply. The detection limit of this system is 0.5 µg benzo(a)pyrene per filter.

Analytical Procedure for Solvent Extraction of Particulate Filters

LARGE SOXHLET EXTRACTION OF ORGANIC SOLUBLES FROM 20x20 PALLFLEX FILTERS

(Condensed Procedure)

Check extraction schedule and obtain filters.

Put solvent and Teflon boiling chips in pot: 400 mℓ measured with graduated cylinder.

Fold filter and place in soxhlet.

Label boiling pot with filter and test number, solvent, and extraction date. Hook-up hoses to condenser, turn on water.

Turn on heaters to large soxhlets to 100%.

---

(1) Swarin, S.J., "Liquid Chromatographic Determination of Benzo(a)pyrene in Diesel Exhaust Particulate: Verification of the Collection and Analytical Methods," General Motors Research Laboratories report GMR-3127 prepared for presentation to the Fourth International Symposium on Polynuclear Aromatic Hydrocarbons, Columbus, Ohio, October 4, 1979, and for publication in "Polynuclear Aromatic Hydrocarbons," Raven Press, 1979.

Record filter data in log book.

Extract for 8 hours - Extractions started by 9 a.m. are completed at 5 p.m.

Filter extracts of 20x20 filters through 47 mm Pallflex filters.

Roto evaporate extract to about 20 mℓ.

Transfer extract to 30 mℓ beaker.

Blow down solvent in desiccator to 2-3 mℓ.

Transfer extract to preweighed sample vial. NOTE: SAMPLE VIALS SHOULD NOT BE TOUCHED WITH BARE HANDS; FINGERPRINTS MAY CAUSE WEIGHING ERRORS. USE TWEEZERS OR WEAR GLOVES WHEN HANDLING.

Dry extract in desiccator.

Weigh vial with cap on.

Record weight in log book.

Calculate percent extractables.

Make a copy of data in log book and give to Mary Ann Warner.

The organic solubles extraction procedure is a very precise method requiring careful, repetitive lab technique. It is necessary in this involved procedure that each step is carried out in a precise and reproducible fashion. If a spillage or other accident occurs while handling a sample, the problem should be recorded in the log book. In this way unexplained irregularities in the results can be traced to the extraction process.

Special attention also needs to be directed to the bookkeeping required for the extractions. An extraction schedule is posted weekly designating which filters are to be extracted. Each soxhlet needs to be labeled with filter and test number, solvent and extraction date. When the extractions are finished, the beaker used in the blowdown step should also be labeled with filter and test number, solvent and extraction date. The weighed sample bottles should be numbered and this number recorded in the log book. After the final weighing, the sample vial should also be labeled. On a <u>daily</u> basis the filters extracted need to be recorded in the log book.

The purpose of the soxhlet extraction is to remove organic soluble material from exhaust particulate collected on filters. Several safety precautions need to be taken when working with diesel exhaust products. The concentrated exhaust products should be treated as potentially hazardous compounds. The following safety precautions are recommended:

1. The soxhlets are set up under a vented hood in case vapors escape from the extractors. Any other step which involves open containers of solvent (sample transfer, filtration, etc.) should be carried out under the hood. Avoid breathing solvent vapors. When the desiccator is opened, a high concentration of solvent vapor is usually emitted. In this case, the door to the desiccator should be opened wide and the vapors allowed to vent into the hood. When the vapor concentration has decreased, work can be continued with the desiccator.

2. Where reasonable, protective clothing should be worn (lab coat and plastic gloves). However, plastic gloves should not be allowed to contact the particulate or organic extractables.

3. If extract spills on the skin, immediately wash off with soap and water. Otherwise, wash hands thoroughly after working with the extracts and before eating or drinking.

LIST OF EQUIPMENT

Extraction

soxhlet - 55/50 top joint, 24/40 bottom joint
condenser - 55/50 joint
500 mℓ flat bottomed boiling pot - 24/40 joint
six position heating mantle
Teflon boiling chips
drying tubes filled with molecular sieve and silica gel

Filtration

Millipore all glass filtration apparatus:
- 300 mℓ cup
- vacuum adapter with glass frit - 40/35 joint
- 1 ℓ filter flask - 40/35 joint
- clamp

vacuum source

Concentration

Buchler flash evaporator with cold finger condenser
1 ℓ round bottom flask
steam bath
hot plate
vacuum source

Drying

desiccator box
gas manifold with curved syringe needles
dry zero nitrogen
aluminum blocks
weighing vials

Accessories

glass petri plates
Teflon squeeze bottles with solvents
transfer pipets plus rubber bulbs
beakers
large and small tweezers
vacuum grease (for condenser hose only)

Chemicals

Extraction
Burdick and Jackson glass distilled cyclohexane, methylene chloride, acetonitrile
Concentration
isopropyl alcohol, dry ice

WASHING PROCEDURES

Glassware

See attached procedure.

Teflon Boiling Chips

Used boiling chips are washed with methylene chloride in the sonic bath. They are placed in a petri dish, covered with methylene chloride and sonicated for about 15 min. The methylene chloride is discarded and fresh solvent is added. The chips are again sonicated for 15 min. The solvent is discarded. If particulate remains on the chips, they are rinsed with methylene chloride until clean. The clean chips are dried under the hood and placed back in the bottle.

Weighing Vial

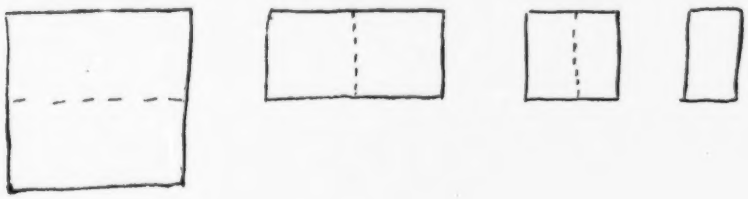
New vials are rinsed in methylene chloride, air dried and stored in trays. Used bottles are solvent rinsed and washed as glassware. Before using again they are rinsed in CH_2Cl_2, air dried and stored in trays. Once the weighing vials have been cleaned do not touch with hands as fingerprints will add weight to the vials. Wear gloves or use tongs when handling vials.

LARGE SOXHLET EXTRACTION PROCEDURE

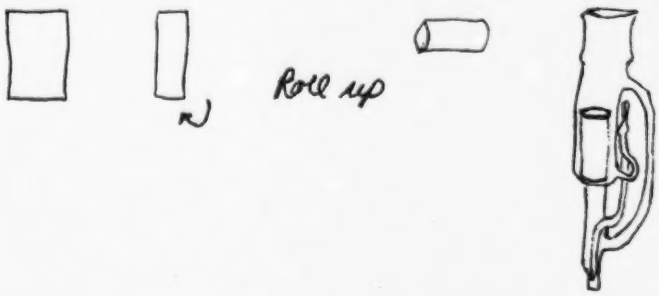
Extraction of 20x20" Pallflex Filters

1. Several (8-10) Teflon boiling chips are dropped into a round, 500 mℓ, flat bottomed flask. The desired Burdick and Jackson glass distilled solvent is added to the flask (400 mℓ of cyclohexane, methylene chloride or acetonitrile) using a 500 mℓ graduated cylinder. 20x20 filters can be found in Tedlar bags in the freezer in the shop. Remove the needed filters and cross through the corresponding filter numbers on the bag. Purge the bag with dry zero nitrogen (same cylinder used to dry organic extracts in the chem lab) and then heat seal the bag. Place bag back in the freezer and lock the freezer. The person weighing filters can help locate filters if they can not be found.

2. The 20x20 filter is then folded as shown below. The filter is usually folded three times before it is placed in the paper envelope.



The filter needs to be folded to a smaller size to fit into the soxhlet below the solvent level. Clear an area and lay out a terry towel to work on. Only metal tweezers should be allowed to contact the filter in the folding process. Do not touch with hands or gloves.



Soxhlet extractor is assembled. Some solvent in the Teflon squeeze bottle should be squirted on the joint to prevent sticking. Slight twisting of the joint will distribute the solvent around the joint. No Vacuum grease should be used. The soxhlet is secured to the stand over the multiple extraction heater so that the flask sits evenly on the heater. Boiling pots are labeled with filter and test number, solvent and extraction date.

3. Water hoses on the condenser are connected so that water flows from the bottom to the top of the condenser. A small amount of vacuum grease may be used on the hose connectors so that the hoses will slip on and off the condenser. If too much grease is used, however, the hoses may pop loose.

4. A drying tube packed with fresh molecular sieve and silica gel (kept in the chem lab oven) and a Teflon connector is placed on top of each condenser. A supply of drying tubes should be kept on hand in the oven. When the indicator changes from dark blue to pale blue or clear the desiccant needs to be changed.

5. The heater and condenser water are turned on. The heater control is turned to 100%.

6. Once a week the cycling rates should be checked. This is done by timing each soxhlets' cycling period three times after the boiling rate has stabilized (about 2 hours). The soxhlets should cycle at least every 15 minutes for a desired rate of 4 cycles/hour. (Timing is done by first allowing the soxhlet to fill with solvent. One cycle is timed from the point at which solvent begins siphoning until the soxhlet fills again and starts siphoning).

7. The large soxhlet extraction is run for 8 hours (9 am - 5 pm).

8. To shut the system down after 8 hours, the heater is turned off. After the solvent stops boiling (15-30 minutes) the cooling water is also shut off.

Concentration of Extract

1. The next day the boiling pots should be placed in the desiccator to prevent condensation of moisture. The solvent remaining in the soxhlet is poured into the waste container.

2. If the filter is extracted with only one solvent, the filter is removed to a glass petri dish with large tweezers and placed in the desiccator or under the hood until dry. The filter is discarded. If the filter is extracted sequentially, the filter is left in the soxhlet until extracted with the last solvent. Then the filter is removed with large tweezers to a glass petri dish and placed in the desiccator or under the hood until dry. The filter is discarded.

3. The pan with approximately 1-1 1/2" of deionized water is heated on the hot plate to 40°C for methylene chloride and to 80° for cyclohexane and acetonitrile.

4. Isopropyl alcohol is added to the cold finger condenser until it is half full. Small chunks of dry ice are slowly added to the alcohol (one at a time, otherwise the alcohol boils over), until the mixture is about 2" from the top. It may be necessary to place a towel on top of the cold finger to prevent splashing.

5. Filtration - All extracts are filtered through a 47 mm Pallflex filter as follows: Filtration apparatus and filter are washed with solvent. The solvent is discarded. The pressure relief valve on the vacuum pump should be fully opened to provide minimum vacuum on the filtration apparatus. The extract plus boiling chips are poured into the filter funnel keeping the liquid level less than half full. Solvent is used to wash the boiling pot and filter funnel three times. Vacuum is turned off as soon as all the liquid is filtered to prevent moist air from being drawn through the filter flask containing the extract.

6. The filtered extract is poured into a prewashed 1000 mℓ round bottom flask with washings. The flask is attached to the roto- evaporator so that the flask is submerged about 1/2" in the pan of heated deionized water. The vacuum is connected to the condenser. The pressure relief valve is closed so that maximum vacuum is pulled (as close to 76 cm Hg as possible).

7. The vacuum pump and roto- evaporator are turned on and allowed to run (approximately 20-40 minutes) until there is only about 20 mℓ of sample remaining in the flask. If the entire amount of solvent is evaporated by mistake, solvent is drained back into the 1000 mℓ flask with washings and re-evaporated to approximately 20 mℓ. The carbon in the vapor filter should be changed about once a month. The micron filters should also be serviced monthly by sonicating the filter frit in methylene chloride for about 30 minutes.

8. The concentrated extract is transferred to a 30 mℓ beaker with washings. The extract is further concentrated by blowing dry zero nitrogen over the sample in the desiccator until the volume is reduced to 2 or 3 mℓ. (The nitrogen flow should be maintained at a rate that does not blow sample out of the beaker or vial). The contents are again transferred with washings to a preweighed sample bottle. NOTE: SAMPLE VIALS SHOULD NOT BE TOUCHED WITH BARE HANDS; FINGERPRINTS MAY CAUSE WEIGHING ERRORS. USE TWEEZERS OR WEAR GLOVES WHEN HANDLING. The extract is completely dried in the desiccator (1-2 hours). Sometimes the sample will appear oily or wet. The extract can be weighed several times to determine if the sample is losing weight to solvent evaporation. If the weight remains stable, the sample is dry and the weight can be recorded. Wooden bottle trays or plastic tubing should not be used in the desiccator as they may contaminate the samples. The molecular sieve and silica gel desiccant should be changed when the indicator turns pale blue or clear. The lifetime of the desiccant can be prolonged if the gas exit valve is closed off overnight. This prevents moist room air from backing up into the desiccator.

9. After the final weighing, the vials are sealed with Teflon tape and labeled.

10. Ames extracts are sealed in a nitrogen purged Tedlar bag and stored upright in dry ice. All other extracts are stored upright (to prevent leakage) in trays.

III. SUMMARY OF RESULTS

The data reported for this work assignment included percent organic extractables (as a fraction of total particulate collected), and in some cases, micrograms of benzo(a)pyrene per milligram of extract. The results are found in Tables 1 through 5. Some samples required additional analyses not included in this program. These additional data and the Ames data are found in the final reports of the respective projects. The reports are footnoted in Tables 1 through 5.

TABLE 1. EXTRACTION DATA FOR FILTERS RECEIVED FROM EPA

Sample Description	Sample Number	Date Received	Particulate Weight (mg)	Total Particulate Weight (mg)	Extract Weight (mg)	Percent Extract-ables	Sample Analysis: Ames Required	Ames Date Shipped	BaP Required	BaP µg BaP/mg Extract
Batch 1										
8-20 x 20 filters	81-0010	4-8-81	550							
10 filter socks	81-0020	4-8-81	330	1230	110.971	9.02	yes	5-20-81	yes	0.0201
from salt mine	81-0030	4-8-81	350							
	81-0040	4-8-81	1040	1040	171.283	16.47	yes	5-20-81	yes	0.0330
	81-0050	4-8-81	230							
	81-0060	4-8-81	460	1230	73.376	6.00	yes	5-20-81	yes	0.0155
	81-0070	4-8-81	540							
	81-0080	4-8-81	30	1030	39.095	3.80	yes	5-20-81	yes	<0.07
	10 socks* (unnumbered)	4-8-81	none provided	--	29657.3	--	yes	5-20-81	yes	not measurable
Batch 2										
19-20 x 20 filters	81-0440	5-26-82	610							
	81-0450	5-26-82	720	2150	746.070	34.70	yes		no	--
	81-0460	5-26-82	820							
	81-0500	5-26-82	330							
	81-0510	5-26-82	280	920	68.760	7.47	yes		no	--
	81-0520	5-26-82	310							
	81-0560	5-26-82	220							
	81-0570	5-26-82	360	1230	647.821	52.67	yes		no	--
	81-0580	5-26-82	420							
	81-0590	5-26-82	230							
	81-0660	5-26-82	460							
	81-0670	5-62-82	450	1380	218.936	15.86	yes		no	--
	81-0680	5-26-82	470							
	81-0690	5-26-82	400							
	81-0700	5-26-82	340	1120	140.719	12.56	yes		no	--
	81-0710	5-26-82	380							
	81-0720	5-26-82	60							
	81-0730	5-26-82	50	170	25.537	15.02	yes		no	--
	81-0740	5-26-82	60							

*Socks extracted in pairs and extracts combined.

TABLE 2. EXTRACTION DATA FOR VOLVO TD100A (DUAL FUEL) AND VOLVO TD100C (DIESEL) FILTERS[a]

Filter A[b] (5830.6-P20-)	Filter B[b] (5830.6-P20-)	Total Part. Weight (mg)	Total Ext. Weight (mg)	Percent Extractables	Sample Analysis: Ames Required	Ames Date Shipped	BaP Required	BaP µg BaP/mg Extract	Sample[c] Processing
			VOLVO TD100A TRANSIENT METHANOL AND CATALYST						
48	--								C
67	--	241.4	38.303	15.87					C
68	--								C
202	--								C
203	--	190.1	41.591	21.88					C
204	--								C
50	--	81.5	17.278	21.20	NO	--	YES	0.0055	C
51	--	77.3	15.837	20.49					C
196	--								C
197	--	289.3	33.749	11.67					C
198	--								C
			VOLVO TD100A TRANSIENT ETHANOL						
136	--								C
137	--	257.3	126.318	49.09					C
138	--								C
127	--				NO	--	YES	0.0017	C
128	--	247.9	126.014	50.83					C
129	--								C
			VOLVO TD100A TRANSIENT ETHANOL AND CATALYST						
145	--								C
146	--	288.8	44.731	15.49					C
147	--								C
184	--				NO	--	YES	0.0921	C
185	--	413.1	34.658	8.39					C
186	--								C
			VOLVO TD100A TRANSIENT METHANOL						
15	--								E & C
4	5	657.4	501.469	76.28	YES	5-13-81	YES	0.0061	E & C
9	10								E & C
11	12								E & C

TABLE 2 (CONT'D). EXTRACTION DATA FOR VOLVO TD100A (DUAL FUEL) AND VOLVO TD100C (DIESEL) FILTERS

Filter A[b] (5830.6-P20-)	Filter B[b] (5830.6-P20-)	Total Part. Weight (mg)	Total Ext. Weight (mg)	Percent Extractables	Sample Analysis: Ames Required	Ames Date Shipped	BaP Required	BaP µg BaP/mg Extract	Sample[c] Processing
			VOLVO TD100C TRANSIENT DIESEL						
311	--								
333	334	161.1	56.267	34.82	YES	5-13-81	YES	0.0168	E & C
335	336								E & C
337	338	901.7	276.459	30.66					E & C
									E & C
			VOLVO TD100A STEADY STATE METHANOL[d]						
238	--	743.5	678.983	91.32					C
212	--								C
213	--	463.9	155.754	33.57					C
214	--								C
219	--								C
220	--	438.8	27.334	6.23					C
221	--								C
232	--	367.9	299.095	81.30	YES	5-13-81	YES	0.0043	C
225	--								C
226	--	230.1	49.422	21.48					C
227	--								C
208	--								C
209	--	390.1	100.508	25.76					C
210	--								C
244	--	753.1	627.469	83.32					C
			VOLVO TD100A STEADY STATE ETHANOL AND WATER[d]						
290	--	1057.3	1029.645	97.38					C
295	--	145.5	58.911	40.49					C
304	--	146.2	32.936	22.53					C
232	--	367.9	299.095	81.30					C
308	--	117.0	17.590	15.03	YES	5-13-81 and 5-18-81	YES	0.0064	C
298	--	202.0	37.831	18.73					C
292	--	1274.6	925.677	72.62					C
307	--	118.1	16.402	13.89					C

TABLE 2 (CONT'D). EXTRACTION DATA FOR VOLVO TD100A (DUAL FUEL) AND VOLVO TD100D (DIESEL) FILTERS

Filter A[b] (5830.6-P20-)	Filter B[b] (5830.6-P20-)	Total Part. Weight (mg)	Total Ext. Weight (mg)	Percent Extractables	Sample Analysis: Ames Required	Ames Date Shipped	BaP Required	BaP µg BaP/mg Extract	Sample[c] Processing
			VOLVO TD100A STEADY STATE METHANOL AND CATALYST[c]						
247	--	842.9	779.919	92.53					C
254	--	383.5	21.104	5.50					C
271	--	671.3	8.325	1.24					C
277	278	97.6	64.805	66.40					E & C
264	--	780.6	25.925	3.32					C
262	--	534.8	16.635	3.11	YES	5-13-81	YES	0.0011	C
249	--	428.9	243.940	56.88					C
252	--	373.4	18.922	5.07					E & C
272	273	1207.2	9.542	0.80					E & C
265	--	908.4	7.210	0.80					E & C
259	263	1129.6	22.462	2.00					E & C
			VOLVO TD100C STEADY STATE DIESEL[d]						
340	--	339.0	303.526	89.54					E & C
349	--	245.0	133.437	54.46					E & C
357	358	318.3	21.642	6.80					E & C
347	--	135.1	109.444	81.00	YES	5-13-81	YES	0.0032	E & C
354	355	456.4	42.238	9.25					E & C
351	352	672.1	65.478	9.74					E & C
344	--	447.8	390.951	87.30					E & C
359	--	158.3	11.422	7.22					E & C
324	--	163.0	64.262	39.42	NO	--	NO	--	E
310	--	158.0	57.353	36.30	NO	--	NO	--	E
330	--	149.8	46.120	30.79	NO	--	NO	--	E

[a] Additional information on these filters can be found in EPA report EPA 460/3-81-023.
[b] Filter nubmers in both Filter A and Filter B columns indicate that two filters were extracted per soxhlet.
[c] C-combined, E-extracted
[d] Ames bioassay will be performed on the extracts from each individial modal cycle in addition to the composite sample.

TABLE 3. EXTRACTION DATA FOR VOLKSWAGEN RABBIT DIESEL FILTERS COLLECTED UNDER AN ALTERNATE FUEL STUDY[a]

FILTER A (5830.3-P20-)	FILTER B (5830.3-P20-)	TOTAL PART. WEIGHT (mg)	TOTAL EXTRACT WEIGHT (mg)	PERCENT EXTRACTABLES	SAMPLE ANALYSIS[b] AMES REQUIRED	AMES DATE SHIPPED	BaP REQUIRED	BaP DATE SHIPPED	SAMPLE PROCESSING[c]
78	--	593.6	144	24.3					E, C&S
79	--	449.5	116	25.9					E, C&S
101	--	788.0	122	15.5					E, C&S
102	--	653.1	74	11.4					E, C&S
90	91	846.0	230	27.2					E, C&S
92	93	590.6	144	24.4					E, C&S
80	--	606.4	145	23.9					E, C&S
103	--	984.6	127	12.9					E&S
94	--	851.0	140	16.4					E&S
82	--								E&S
83	--	1238.2	180	14.6					E, C&S
48	--								
49	--	1654.6	245	14.8					E, C&S
57	--								
58	--	1356.6	184	13.6					E, C&S
69	--								
70	--	1747.8	263	15.0					E, C&S
72	--								
73	--	1182.8	296	25.0					E, C&S
98	--								
99	--	1437.5	298	20.7					E, C&S
107	--								
108	--	1656.8	241	14.6					E, C&S
111	--								
112	--	1510.1	192	12.7					E, C&S

[a] Additional information on these filters can be found in EPA Report, EPA 460/3-82-002.
[b] Various analyses performed under another task of this project and published in above mentioned report.
[c] C-combined, E-extracted, S-split

TABLE 4. EXTRACTION DATA FOR TWO FORD ESCORTS AND TWO VW RABBITS USING SEVERAL COMBINATIONS OF FUEL AND CATALYST[a]

Filter A[b]	Filter B[b]	Total Part. Weight (mg)	Total Extract Weight (mg)	Percent Extractables	Sample[c] Processing
(5830.11-P20-) 9 11	(5830.11-P20-) 10 12	160.6	46.559	28.99	E,C
13 15	14 16	270.5	47.955	17.73	E,C
25 27	26 28	198.3	50.955	25.72	E,C
37 39	38 40	227.3	58.718	25.83	E,C
41 43	42 44	177.4	52.485	29.59	E,C
(6619-P20-) 17 19	(6619-P20-) 18 20	325.6	54.060	16.60	E,C
77 79	78 80	2807.5	30.619	1.09	E,C
(6619.3-P20-) 57 59	(6619.3-P20-) 58 60	198.6	56.305	28.35	E,C

[a] Additional information on these filters will be published in the final report of another phase of this project: Contracts 68-03-2884, Task Specifications 11 and 12 and 68-03-3073, Work Assignments 1 and 3.

[b] Benzo(a)pyrene and Ames analyses not required on this Work Assignment.

[c] Filter numbers in both Filter A and Filter B columns indicate that two filters were extracted per soxhlet.

[d] E-extracted, C-combined

TABLE 5. EXTRACTION DATA FOR A MACK EM6-300 HEAVY-DUTY DIESEL ENGINE USING ALTERNATE FUELS[a]

Filter A[b] (5830.14-P20-)	Filter B[b] (5830.14-P20-)	Total Part. Weight (mg)	Total Extract Weight (mg)	Percent Extractables	Sample Analysis: Ames Req'd	Ames Date Shipped	BaP Req'd	BaP µg/BaP Sample	Sample Processing[c]
FUEL 509									
13	14	625.8	133.028	21.26	Yes	6-29-82	Yes	0.0631	E,C
15		330.8	66.871	20.21					E,C
19	20	601.1	118.703	19.75					E,C
21	16	1224.0	230.601	18.84	Yes	6-29-82	Yes	0.0099	E,C
17	18								E,C
FUEL 510									
40	41	1031.5	773.070	74.95	Yes	6-29-82	Yes	0.2045	E,C
42		559.7	386.898	69.13					E,C
43	44	663.0	389.045	58.68					E,C
45	46	1374.0	754.077	54.88	Yes	6-29-82	Yes	0.1425	E,C
47	48								E,C
FUEL 511									
22	23	855.1	129.543	15.15	Yes	6-29-82	Yes	0.2969	E,C
24		403.1	71.888	17.83					E,C
25	26	1157.2	264.663	22.87					E,C
27	28				Yes	6-29-82	Yes	0.0361	E,C
29	30	542.5	126.768	23.37					E,C
FUEL 517									
31	32	873.5	119.248	13.65	Yes	6-29-82	Yes	0.0750	E,C
33		438.0	58.837	13.43					E,C
34	35	692.2	114.419	16.53					E,C
36	37	1371.4	218.856	15.96	Yes	6-29-82	Yes	0.0479	E,C
38	39								E,C

[a] Additional information on these filters will be published in the final report of another phase of this project: Contract 68-03-2884, Task Specification 14.

[b] Filter numbers in both Filter A and Filter B column indicate that two filters were extracted per soxhlet.

[c] E-extracted, C-combined

APPENDIX A

SCOPE OF WORK

Contract 68-03-2884
Task Specification 9

SCOPE OF WORK

The purpose of this task is to provide ECTD with extraction and BaP analysis support. This will consist of a number of work elements, including: performing soxlet extractions on a given set of filters, determining the proportion that is solvent soluble organic, performing BaP analysis, mixing the appropriate filter extracts to form a sample, taking the sample to dryness, shipping the sample under dry ice to ECTD's Ames test contractor, EG and G Mason Research. The exact instructions for each sample may vary and will be given upon sending the samples to SwRI. Most Ames Test Samples will consist of a combination of extracts from two or more filters. Under most conditions, the filters can be extracted in groups, providing the apparatus is adequately large. There will be some specific cases where this may not be done; for example, when individual BaP or Ames Test analyses must be done. The following is a Table of the expected sample requirement:

Project	No. of Ames Samples	Estimated No. of Filters	Estimated No. of BaP Analyses	Time Frame Required, 198
SDSB-H.D. Diesel Baseline	3	21	0	Jan-March
SDSB-Misc.	10	20	10	Jan-March
TAEB-Trap Testing	15	45	0	Jan-Dec.
TAEB-Misc.	10	25	0	Jan-Dec.
CTAB-L.D. Diesel Organics	12	24	0	Sept-Jan.
CTAB-Volvo	8	56	8	Jan-April
CTAB-H.D. Diesel Malfunction	4	28	14	Feb-May
CTAB-Synthetic Fuels (VW)	10	20	10	Feb-Dec.
CTAB-Misc.	10	20	7	Jan-Dec.
TOTAL	82	239	49	

The reporting requirements shall consist of: 1) monthly progress letters outlining the progress of the work as well as the resulting data, and 2) a final letter report briefly outlining the conduct of the task.

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